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APPLICATION OF NANOPARTICLES BIO-SYNTHESIZED FROM ASOKA *(POLYALTHIA LONGIFOLIA)* LEAVES ON COTTON TEXTILES

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ABSTRACT

As textile materials are next to skin, the problem of infestation is aggravated due to the suitable climate provided by the skin, in turn leads to multiplication of microorganism. So, in the present study an attempt was made to finish cotton textiles with bio-synthesized Polyalthialongifolia leaves Nanoparticles to impart antimicrobial finish. An effective and environment friendly technique called, green synthesis/biosynthesis of Asoka leaves extract was carried out from 1mM of $AgNO_3$.Nanoparticles when analyzed under UV- spectrophotometer, have shown wave length of 418nm with an absorbance of 2.56suggesting the formation of Nanoparticles. Through TEM spherical Nanoparticles was observed with51.8nm sizes.

Woven and knitted cottons were applied with 5 percent Nanoparticles with; padding & drying at room temperature (T1) and a pad-dry-cure method at 80^0 C (T2) treatments. Treated fabrics were assessed for its antimicrobial activity following AATCC 147-1998 for antibacterial activity against both E. Coli and S. Aureus and AATCC 30-1993 test method for antifungal activity. Amongst all the samples,T1 treated samples gave better results than T2 treated samples. Even after five to ten launderings pad-dry-cure samples showed substantial antibacterial property. Analysis of geometrical parameters of fabrics showed an increase in yarn count, fabric count, thickness and fabric weight.

KEYWORDS: Asoka (Polyalthialongifolia) Leaves, Bio-Synthesis, Nanoparticles, Cotton Textiles, Antimicrobial Finish

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Original Article

INTRODUCTION

Cellulose content of the cotton material provides basic requirements such as moisture, oxygen, nutrients and temperature for bacterial growth and multiplication. This often leads to objectionable odor, dermal infection, product deterioration, allergic responses and other related diseases, which can be avoided, if the cotton fabrics are treated with antimicrobials. As cotton is most widely used textile material for its properties like absorbency, comfort, etc. these materials require good microbial resistance from different microorganisms. With environmental awareness and importance on organic sources, attention has been focused on plant materials as a source of antimicrobials. Natural antimicrobial agents are non-toxic and non-allergenic and do not cause the problems of microbial resistance (Gupta and Bhaumik, 2007). Attempts have been made to introduce these plant sources onto textile materials. Use of natural sources like plants and metals such as silver as antimicrobial material has been practiced since ancient times in folk medicine. Experiments conducted in these lines showed few difficulties such as color change, stiff hand and loss in fabric strength due to the use of these materials in their raw form. Further, it imparted only a renewable finish to the textile materials. Hence, scientists extended their experiments using different parameters to stabilize the antimicrobial property on textiles for longer period. As a result attention was

paid to reduce the size and composition of these plant sources to overcome these difficulties. The answer to this is to reduce the plant material to nano sized particles. As materials at nano level impart excellent properties to the materials used. Nano particles exhibit completely new or improved properties based on specific characteristics such as size, distribution and morphology. Patel & Chatopadhyay (2007), in there article have specified Nanoparticles usage in textiles can the properties of the textile materials. In this present study, an attempt has been made to finish cotton woven and knitted textiles with biosynthesized Asoka (*Polyalthialongifolia*) leaves nanoparticles to impart antimicrobial finish.

METHODS AND MATERIALS

Preparation of Plant Sources and Cotton Fabrics

Asoka (*Polyalthialongifolia*) leaves were collected from the study area, which were cleaned with ethyl alcohol (ethnol) along with distilled water in 1:5 ratio. Cleaned leaves were tray dried at 50^0C. Dried leaves were powdered and sieved with flour sieve to obtain fine powder. Later it was stored aseptically.

Considering the wide usage of woven cotton and semi bleached knits as under garments material, they were sourced from Hyderabad and Tirupur respectively. Dirt, oil and other impurities of woven were removed through scouring process, where semi-bleached knitted material was soaked in water for 2hrs.

Synthesis of Asoka Leaves Nanoparticle Synthesis

Powder prepared from Asoka leaves were soaked in distilled water and was decanted. Mix obtained decant with 1mM solution of AgNO3 in 1:9 ratio. The color changed broth was centrifuged at 18,000 RPM for 25-30 minutes for nanoparticles formation.

Analysis of Nanoparticles for its Morphology

UV – Visible Spectrophotometer

Surface Plasmon Resonance of Nanoparticles formation was analyzed through UV – Visible Spectrophotometer with a wave length on X-axis and absorbance on Y-axis.

Transmission Electron Microscopy (TEM)

The size of nanoparticles was analyzed through Flotation method using Transmission Electron Microscopy at 30 magnificence. The sample was tested at Ruska lab, SVVU, Hyderabad

Nanoparticles Finish on Cotton Textiles

Padding technique was used to apply5 per cent synthesized nanoparticles to woven and knitted cottons at 70per cent pressure with MLR of 1:10. One set of samples were shade dried at room temperature are coded as T1, where other were pad-dry-cured at 800C for 15 minutes are coded as T2.

Analysis of Treated Samples for Antimicrobial Efficiency

In the present research, under antimicrobial tests, antibacterial and antifungal efficiency of the treated samples were analyzed. These tests were developed primarily to determine the effectiveness of antimicrobial finishes (Billie and Helen, 1999).

Antibacterial activity, AATCC-147, 1998

For antibacterial activity AATCC-147, 1998 test method was used to test against *Escherichia Coli* and *Staphylococcus Aureus*. The growth rate of culture was evaluated based on the 'Inhibition Zone' or 'Zone of Inhibition' (zoi) of the test sample (Nimittrakoolchia, 2009).

Antifungal Activity

For antifungal assessment, Agar diffusion test - AATCC 30-1993 and Mycelial growth test methods were used.

AATCC 30-1993, Agar Diffusion Test

To test antifungal activity through AATCC 30-1993, the test sample was immersed in the prepared PD broth for 3 days. After 3 days the test tube was observed for the growth of fungi, which was rated as 1- No growth, 2- Minimum growth, 3- Moderate growth & 4-Maximum growth (RajKumar and Krishnaveni, 2007).

Mycelial Growth Test

Mycelial growth on the test sample placed in PD agar petri dish was measured after 1, 3 and 5 days(RajKumar and Krishnaveni, 2007). By observing the mycelial growth the effectiveness of the finish can be determined.

Fabric Testing

Preparation of Test Specimen

The test specimen was cut according to the standard template depend on the type of the test to be conducted. All the creases and 10 centimeters from the selvedges are avoided while cutting the test specimen. Depend on the type of test replicates were cut in such a manner that, no two samples were cut in the same set of warp or weft yarns.

Atmospheric Conditions

To maintain the moisture equilibrium of the test sample, it was conditioned for 24hrs at 27 ± 2^0C with a standatd atmosphere of 65 ± 2 percent relative equilibrium.

Geometrical Parameters

Yarn Count

To determine the fineness of yarn, indirect method was used for counting the number of yarns per unit mass with Beesley's balance, Booth, 1983. This instrument works on the principle of the fixed weight and length. Four samples of the fabric both warp and weft ways were cut separately using the template.

Fabric Count

Under zero tension and free from the folds and wrinkles, count in woven fabric and knitted fabrics is the number of warp & filling yarns; and the number of wale and course loops per unit distance of inch respectively (ASTM 2007). IS1963 – 1969 test method was used to test the fabric count with pick glass having a magnifying lens and a pointer which traveled along the graduated base.

Fabric Thickness

To measure the thickness of the fabric, Heal's thickness gauge was used (Booth, 1983). By laying the fabric flat on the bottom plate and applying a known arbitrary pressure by the upper plate, the distance between one surface and its opposite(ASTM, 2007) of the fabric was measured in micrometers.

Fabric Weight

Following IS No. 1964-1970, the weight of the specific size of the test sample was measured. A circular specimen of 100 Cms^2 which is exactly $1/100^{th}$ of a square meter was cut and weighed by using sensitive balance capable of weighing to an accuracy of 0.001mg.

All the samples were laundered for 1, 5 and 10 times and tested for antimicrobial and geometrical properties at each stage.

RESULTS

Weight of Asoka Leaves at Different Processing Levels

After drying for 4hrs leaves shown 58.33 per cent reduction in its weight, where after grinding and sieving it's further reduced to54 percent, as depicted in Table 1. Later this powder was used for synthesis of nanoparticles.

Table 1: Weight of Leaf Sources at Different Stages (g)

Fresh Leaves	After Drying	Powder after Sieving
600	250	115

Analysis of Nanoparticles

UV – VIS Spectophotometery

During synthesis, extracted leaf solution when subjected with Silver ions in aqueous medium, exhibited color change from mustard to dark brown which was due to Plasmon resonance phenomenon. The results showed the potentiality of plant source to be synthesis into Nanoparticles with $AgNO_3$.UV – VIS spectrograph of Nanoparticles has recorded peak as 418nm with 2.56 absorbance. Broadening of peak as shown in Figure 1 indicate that the particles are polydispersed. Similar peaks were found when Nanoparticles were synthesized from papaya fruit extract with a peak of 450nm by Jain *et al. (*2009).

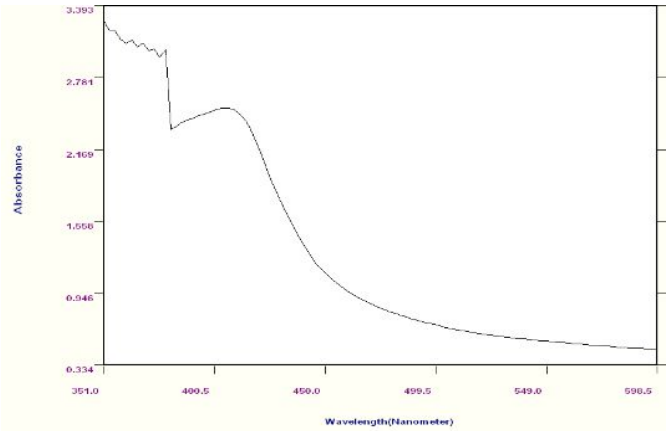

Figure 1: UV – VIS Absorption Spectra of Synthesized Asoka Leaves Nanoparticles

Transmission Electron Microscopy (TEM) Analysis

Synthesized Nanoparticles were scanned through TEM at 30 magnification and it was found that, the Nanoparticles were spherical in shape, as shown in Figure 2. with a size of 51.8nm. All the Nanoparticles were well dispersed.

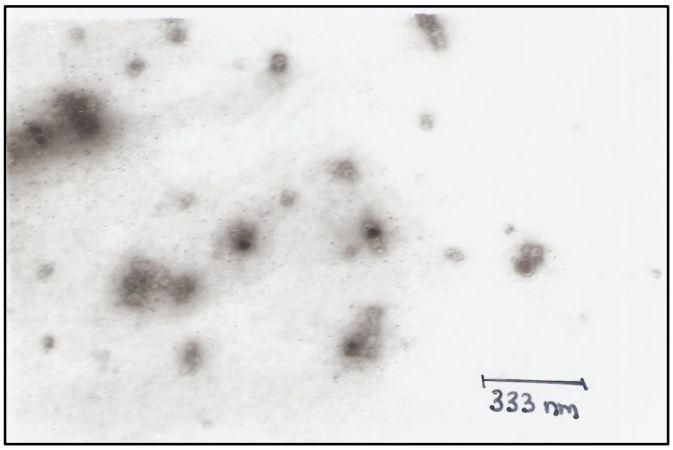

Figure 2: TEM Images of Synthesized Asoka Leaf nanoparticles

Antimicrobial Analysis

Untreated and treated samples were subjected to different antibacterial and antifungal test methods to determine antimicrobial properties of the fabric.

AATCC 147-1998 – Antibacterial Analysis

zoi describes the efficiency of the antibacterial property in the tested substrate and it is the area around the treated substrate into which the antimicrobial chemistry leaches or moves, to kill or inhibit microorganisms. (Kavitha *et al*. 2006).

Antibacterial Assessment of nanoparticles against *S. Aureus* and *E.Coli*

Asoka leaf Nanoparticles showed *zoi* of around 6 to 12mm, where *S.aureus* showed better results than *E.coli*, as shown in. According to Chanda and Nair, 2010 antibacterial activity of *Polyalthialongifolia (Sonn) leaves* was more pronounced against gram positive bacteria. This may be due to the layer of peptidoglycan in gram positive bacteria being (about 20-30 nm) thicker than gram negative bacteria (Silver *et al*. 2006).

Antibacterial Assessment of Untreated and Treated Samples against *S. Aureus* and *E.Coli*

Unfinished woven and knitted samples did not showed any *zoi* against both gram negative and gram positive bacterial cultures, which indicated that the raw fabric did not possesses any antibacterial activity.

The antibacterial effects of nanoparticles prepared by plant sources obey a dual action mechanism of antibacterial activity i.e., the bacterialcidal effect of metal ion present in nanoparticle and membrane disrupting effect of the polymer subunits (Jain *et al*. 2009).Antibacterial activity of woven and knitted samples with T2 has shown more *zoi* than T1, for both the cultures. Among all the treated samples, woven and knitted T2 sample has more *zoi* for *gram positive culture*, however knitted T2 sample have registered maximum *zoi* as 5nm, as shown in Table 2. The antibacterial activity was more pronounced for *gram positive* bacteria than *gram negative* bacteria, this may be due to the thicker layer (about 20-30nm) of

gram positive bacteria than gram negative bacteria.

Table 2: Zone of Inhibition of the Treated Samples and Samples After 5 & 10 Launderings against Bacteria (mm)

Treatments	*zoi* against *E.Coli.*				*zoi* against *S.Aureus*			
	Woven		Knitted		Woven		Knitted	
	T1	T2	T1	T2	T1	T2	T1	T2
Treated samples	3.17	3.5	3.35	4.53	2	2.5	3	5
Samples after I wash	1.53	1.25	1.82	3.03	2	2.25	2.57	3
Samples after V wash	-	2	-	2	-	2.5	-	2.25
Samples after X wash	-	2	-	1.5	-	1.5	-	2

Antibacterial Assessment against *S. Aureus* and *E.Coli* of Samples after One, Five and Tenlaunderings

T2 knitted samples have retained good antibacterial activity, where massive difference was observed against *E.coli* in comparison with T1 sample. Compared to *gram negative* culture *gram positive* culture for woven samples have shown great difference in antibacterial activity, as shown in Table 2. There was not much difference was noticed for T2 knitted sample for both the cultures, whereas T2 woven sample is more active against *S.aureus* than *E.coli*. Even when compared T1 woven samples antibacterial activity is more pronounced against *S.aureus* than *E.coli*, as shown in Table 2.

From Table 2, it was clearly noticed that, the samples with repetitive launderings had lowered antibacterial activity. Especially T1 samples did not possess any antibacterial activity after five washings. This phenomenon is associated with the weak physical bonding between the nanoparticles and the fiber polymer system, which in turn effects on the decreased *zoi* for the later tested samples. However, T2 samples have retained 35 to 57 per cent of the antibacterial property after five launderings and 21 to 57 per cent even after ten launderings against *E.coli*.

Antifungal Activity

AATCC 30-1993, Agar Diffusion Test

Assessment of Untreated and Treated Samples for Antifungal Activity

The fungal growth was analyzed on four point scale. After three days, untreated woven fabric showed maximum fungal grown on the top as a layer, whereas test tube with knitted fabric has developed fungus mixed within the broth, which accounts to moderate fungal growth as shown in Table 4. None of the treated samples possess any fungal growth, irrespective of type of the fabric.

Table 4: Fungal Growth Ratings Tested Samples

Parameters	Woven		Knitted	
	T1	T2	T1	T2
Untreated samples	4		3	
Treated samples	1	1	1	1
Samples after I wash	1	1	1	1
Samples after V wash	-	1	-	1
Samples after X wash	-	1	-	1

Assessment of Treated Samples at Different Laundering Cycles for Antifungal Activity

No growth was observed for the T1 and T2 samples for both woven and knitted fabrics, as shown in Table 4. After first laundering no change was observed for T2 woven sample. There was no fungal growth observed for all the treated samples even after five to ten launderings.

Mycelial Growth Observation

Mycelial Fungal growth was observed on first, third and fifth day.

Assessment of Untreated and Treated Samples for Mycelial Growth

On completion of 24hrs untreated woven samples has developed fungus around test sample, was recorded as 0.6mm (shown in Table 5), on third day fungus developed to 1mm, which further grown to three folds at the end of fifth day.

All the treated samples did not shown any mycelial growth even at the end of fifth day. It can be concluded that samples treated with Asoka Nanoparticles gave best results in terms of mycelia growth. It was further noticed that there was no growth of fungus even after ten washes.

Table 5: Mycelial Growth of Unfinished and Treated Samples (mm)

Fabric Type	Samples	I day	III day	V day
Woven Cotton	Untreated	0.6	1	1.75
	T1 treated	-	-	-
	T2 treated	-	-	-
Knitted Cotton	Untreated	-	0.5	1
	T1 treated	-	-	-
	T2 treated	-	-	-

Assessment of One, Five and Ten Times Laundered Samples for Mycelial Growth

The samples were further tested for mycelial growth after first, fifth and tenth laundering. None of the samples exhibited any mycelial growth even after five days. Thereof, it was determined that, the treated samples possesses good potential against mycelial growth.

Fabric Testing- Geometrical Parameters

Yarn Count(s)

There was an increase in the yarn count for both warp and weft ways after the samples were treated with Nanoparticles, as shown in Table 6. This increase in yarn count is may be due to the shrinkage of yarns in the pre-treatment process. Compared to T1 samples, T2 samples has shown pronounced difference in yarn count, this may be due to further shrinkage of yarns caused by bone drying at elevated temperature.

Fabric Count (No. of Yarns per In.)

An increase in warp count was observed for treated samples over untreated ones for woven fabric. No difference was observed in warp direction, where a decrease in fabric count was observed in weft direction for both type of treated samples than untreated samples, shown in Table 6. The number of wales and courses remained same for treated and untreated samples. As the knitted fabrics are compact in structure, it did not showed further shrinkage, resulted in unchanged fabric count.

Fabric Thickness (mm)

Compared to untreated woven fabric, there was an increase in the thickness for both the treated fabrics. This increase in thickness in woven samples may be due to the Nanoparticles embedded within the fabric. T1 sample possess more thickness than T2 samples. This is due to compressed yarns during padding and curing process. Among knitted

samples T1 samples recorded highest thickness, with 0.718mm than T2 samples. This may be due to flattening of yarns and the protruding fibers, a general characteristic of knitted fabric, thereby reducing the thickness of the fabric. The knitted fabrics were thicker than the woven samples due to loosely spun yarn used in fabric, as a result knitted fabric have greater pliability.

Fabric Weight (GSM)

There was almost equal weights observed for treated samples to untreated cotton samples. Whereas, difference was observed for knitted untreated sample to T1 knitted sample. But, T2 knitted sample did not possess any change in its weight, this may be due to the compression of fabric through rollers during finishing with padding mangle.

Table 6: Geometric Parameters of the Test Samples

Parameters		Yarn count (s)		Fabric count (No./In.)		Fabric thickness (mm)	Fabric weight (GSM)
Samples		Warp	Weft	Warp/ Course	Weft/ Wales		
Woven	UT	41.4	36.6	77	56	0.282	0.8162
	T1	43	37	81	53	0.3	0.8088
	T2	45.8	38.8	81	52	0.288	0.8152
Knitted	UT	-	-	37	36	0.709	1.68
	T1	-	-	37	36	0.718	1.8
	T2	-	-	38	36	0.688	1.68

CONCLUSIONS

From the present research, it can be concluded that, Asoka leaf nanoparticles have proven to be good antimicrobial finish on cotton textiles. As untreated samples did not possess any antibacterial property, it would be healthier to finish cotton fabrics with antimicrobial agents to protect one from different types of infestations, obnoxious odors and damages caused to the textile material through deterioration, because of microorganisms built by body exudates and other favorable body conditions. After evaluating all the samples, it is clearly evident that the T2 treatment helped the samples to resist more against both cultures by recording good *zoi*. From the results, it was evident that samples treated with Asoka Nanoparticles have maximum antibacterial activity by possessing around 60 per cent antibacterial retention even after ten wash cycles. Antimicrobial effectiveness by Agar diffusion test method against *E.Coli* (gram negative) and *S.Aureus* (gram positive) cultures, resulted stronger activity for gram positive culture than gram negative culture, which may be due to the thicker peptidoglycan layer more thicker (about 20-30 nm) in gram positive bacteria than gram negative bacteria. As well antifungal activity by both methods have shown positive results for the Nanoparticles treated samples, among which T2 found to be good finish. Compared to UT, there was an increase in the geometrical parameters was observed for Nanoparticles treated samples.

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